Atlantic Richfield Company

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November 13, 2009

Ms. Nadia Hollan Burke Remedial Project Manager U.S. Environmental Protection Agency Region 9 75 Hawthorne Street, SFD-8-2 San Francisco, CA 94105

Subject: Revised VLT Characterization Work Plan Using X-Ray Fluorescence, Yerington

Mine Site, Lyon County, Nevada: Administrative Order on Consent, EPA Docket

No. 09-2009-0010

Dear Ms. Hollan Burke:

Based on comments provided to Atlantic Richfield Company (ARC) by the U.S. Environmental Protection Agency - Region 9 (EPA) on October 30, 2009 on the Draft VLT Characterization Work Plan Using X-Ray Fluorescence (XRF Work Plan) dated October 30, 2009, and subsequent discussions between ARC and EPA on November 5, 2009, ARC has prepared this revised XRF Work Plan. This XRF Work Plan has been designed to support the removal action for a number of former Anaconda evaporation ponds and Sub-Area A located on the Yerington Mine Site (Site) in Lyon County, Nevada. The removal action is required under the Administrative Order on Consent (AOC) and associated Scope of Work (SOW)¹ dated April 21, 2009 (effective May 1, 2009). The SOW states that:

Respondent (ARC) shall provide and implement a design plan to limit the ponding of low pH, metalliferous water in the lined evaporation ponds to the northwest within the Site to prevent to the extent feasible threats to wildlife as determined by EPA. The design plan also shall include proposed measures to limit the migration of dust containing hazardous substances from the lined and unlined evaporation ponds to the northwest within the Site and those areas known as the "Thumb Pond" and the "Sulfide Tailings Area".

Respondent shall submit a design plan that shall include the use of Vat Leach Tails (VLT) fill material. Respondent shall also submit an implementation work plan for the construction of a VLT cap to be installed over the lined and unlined evaporation ponds, including enhancing the deteriorated cap at the Thumb Pond and capping the area identified as Sub-Area A. The thickness of the VLT cap shall be sufficient to mitigate standing water within the lined evaporation ponds, and to mitigate the generation of fugitive dust from the underlying sediments in the lined, unlined, Thumb Pond and Sub-Area A in Figure 1. The average thickness of the VLT cap will be 18 inches. Portions of the VLT cap may be thicker or thinner than 18 inches as appropriate based on engineering and design requirements to meet the stated objectives. Finally, Respondent shall submit a removal action report after completion of the work.

¹ Administrative Order on Consent and Settlement Agreement for Removal Action and Past Response Costs Anaconda Copper Mine, Yerington Nevada; U.S. EPA Region IX; CERCLA Docket No. 09-2009-0010.



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The locations of the Lined and Unlined Evaporation Ponds (LEP and UEP), the Thumb Pond and Sub-Area A are shown on Figure 1. Per the SOW schedule, ARC submitted the *Anaconda Evaporation Ponds Removal Action Characterization Data Summary Report* (RAC DSR) and *Draft Design Work Plan for the Evaporation Ponds Removal Action* (Draft Design Work Plan) to EPA on June 12, 2009 (Brown and Caldwell, 2009a,b). The Draft Design Work Plan describes the VLT covers for the LEP, UEP, Thumb Pond and Sub Area A as having an average thickness of 18 inches. To limit potential accumulations of poor quality water within the LEP, additional VLT fill materials will be placed in low-lying areas of the LEP to contain excess soil moisture.

Subsequent to these submittals, ARC transmitted the *Draft Implementation Work Plan for the Anaconda Evaporation Pond Removal Action* (Draft Implementation Work Plan; Brown and Caldwell, 2009c) dated November 4, 2009 to EPA. The Draft Implementation Work Plan indicates that approximately 580,000 cubic yards of VLT material will be required for the removal action. Figure 2 illustrates the locations of the proposed VLT borrow source areas, as well as the location of the LEP, UEP, and Thumb Pond. Recent particle size distribution testing of the VLT material indicates that it is composed of the following percentages of sand, gravel and fines:

- sand (fine to coarse grained) about 45 percent;
- gravel (fine [3/4-inch minus]) approximately 40 percent; and
- fines (silt and clay particles) remaining 15 percent.

Although the VLT materials are believed to be generally homogeneous, geotechnical and geochemical differences may occur in the spent ore materials given the different oxide ore types mined and processed by Anaconda. For example, moisture contents obtained from geotechnical samples described in the Draft Implementation Work Plan indicate a range of in-situ moisture contents from 4 to 7 percent. Visual observations of VLT materials indicate: 1) localized surface coatings of green-and white-colored copper and aluminum hydroxides; and 2) very localized occurrences of 'iron-bleed tailings' (i.e., 'red dust'), a fine-grained material resulting from the vat leach ore process, occur within the oxide tailings piles. Table 1, reproduced from the RAC DSR (Brown and Caldwell, 2009a), presents available VLT geochemical data (data collected by SRK Consulting on behalf of the Nevada Division of Environmental Protection [NDEP] in support of a previous removal action that used VLT to cover select 'red-dust areas on the Site).

Recent discussions with EPA have determined the need for additional screening of the chemical character of VLT materials to be placed on the ponds and Sub-Area A during the removal action in order to preclude the placement of VLT materials containing concentrations that may exceed a 'to-be-determined threshold' (see below). In order to not adversely affect the construction schedule for the removal action, a field-portable x-ray fluorescence (FPXRF) instrument will be used in accordance with EPA Method 6200 (Attachment 1) to accomplish this objective, as described below.

Work Plan Objectives

The objective of this XRF Work Plan is to determine the correlation of metal concentrations obtained during field screening of VLT materials using a FPXRF instrument with the analytical results for VLT samples obtained from the project laboratory for a subset of the metals presented in Table 2 (XRF instruments do not measure all the metals that can be analyzed in the laboratory). The correlation will be used to provide the basis for screening VLT materials during the removal action. Because XRF detection limits are generally much higher than the detection limits that result from laboratory analyses, ARC anticipates that a correlation between low concentration results will have a much higher degree of uncertainty than for high concentration results and, that for a number of metals, no correlation will be possible. However, it is the higher concentration results that are of interest for the VLT screening during the removal action.

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FPXRF Instrument

The FPXRF instrument selected to perform the field screening of the VLT borrow source areas is an Innov-X Handheld energy dispersive XRF (EDXRF) that uses a powerful, miniature X-ray tube excitation source and a high resolution silicon PIN diode detector (Attachment 2) to provide improved performance and a lower level of detection (LOD) than most other commercially available FPXRF instruments. The Innov-X Handheld XRF has a limit of detection for copper as low as 15 parts per million (ppm), equivalent to milligrams per kilogram (mg/kg). The LODs achievable for various metals using the Innov-X Handheld XRF under interference-free conditions are as follows:

	<u>Metal</u>	LODs (ppm)
•	Antimony	30 – 50
•	Arsenic	7 – 10
•	Barium	150 – 200
•	Cadmium	15 – 20
•	Chromium	30 – 50
•	Copper	15 – 20
•	Lead	10 – 15
•	Mercury	10 – 15
•	Nickel	20 - 25
•	Selenium	7 – 10
•	Silver	15 – 20
•	Thallium	10 – 15
•	Tin	30 - 50
•	Zinc	15 – 20

More information regarding the FPXRF instrument proposed for use in the screening process is provided in Attachment 2. As described in EPA Method 6200 (Attachment 1), the instrument sensitivity and LODs that can be achieved under actual field operating conditions depend on a number of factors including the analyte(s) of interest, the type and strength (i.e., power output) of the excitation source, the type of detector used, the position of the FPXRF during measurements, the measurement duration, physical matrix effects, chemical matrix effects (i.e., interelemental spectral interferences), and moisture effects. Potential errors associated with these factors are not as significant as user- or application-specific factors such as measurement duration, physical matrix effects, chemical matrix effects, and moisture effects. These field-related factors may result in data variability that can adversely affect the correlation between instrument readings and laboratory analytical results. EPA Method 6200 (Attachment 1), describes procedures to minimize the cumulative impact of these potential errors, which will be implemented as part of this XRF Work Plan (see the **Field Screening Procedures** below for additional details).

User-specific factors include separation distance and measurement duration. Potential error increases in direct relation to the separation distance between the instrument and the sample. Therefore, minimizing this error will be accomplished by keeping the FPXRF probe window in direct contact with the sample whenever a measurement is taken. Measurement duration affects the precision and detection limits of each result. Increasing the measurement duration by a factor of four will improve precision by a factor of two and will improve detection limits by 50 percent (EPA Method 6200 – Attachment 1). However, it has been recognized that increasing the measurement duration beyond 120 seconds yields diminishing returns. The following optimization steps will be performed at the first sample location prior to the start of VLT field screening to determine the optimal measurement duration:

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- 1. One of the four quadrants of a flat-lying zip-lock bag containing VLT sample materials will be used for all of the optimization measurements; copper measurements only will be used.
- 2. A series of 10 FPXRF measurements be taken and recorded on a field form at each of four progressively longer measurement durations consisting of 60, 80, 100 and 120 seconds.
- 3. Summary statistics including mean, standard deviation (SD), variance (V), and coefficient of variation (CV) will then be calculated for the 10 FPXRF measurements taken at each of the four measurement durations. SD, V, and CV values calculated for each of the four sets of FPXRF measurements will be plotted versus the corresponding measurement duration.
- 4. A best fit trend line will be fitted to the plotted data points for each parameter (SD, V, and CV), and the approximate inflection point (i.e., the trend line where a change in slope indicates that the measurement duration will no longer affects the measurements) will be identified (if present).
- 5. The measurement duration to be used for subsequent FPXRF field screening will be the longest time associated with the inflection point of the trend line for any of the three plotted statistical parameters (if only one inflection point is identified, that value will be used). If no obvious infection point can be identified, 120 seconds will be used for the FPXRF field screening process.

Physical matrix effects result from variations in the physical characteristics of the sample material. These can include variations in particle size, uniformity, homogeneity, and surface conditions. Because XRF is a surface analytical method (i.e., the measurement only represents conditions on the surface of the sample particles), which is indicative of an approximate one square centimeter area (i.e., a small portion of the overall sample area), XRF can be more sensitive to sample heterogeneity than standard laboratory methods that involve partial or complete digestion of the entire sample before analysis. Given that the variability of XRF results associated with sample heterogeneity would be most significant for in-situ measurements, the most effective approach for minimizing this source of potential error is to use only intrusive sample measurements. Each sample will be evenly distributed in a one-gallon plastic bag before FPXRF measurements are taken to provide uniform surface conditions for the XRF measurements (the bagged sample will be the same sample material being submitted to the analytical laboratory).

Chemical matrix effects result from interactions between fluorescent X-rays from different elements. These effects can occur as either spectral interferences (peak overlaps) or as X-ray absorption and enhancement phenomena. Both effects are common in soils containing heavy metals. One of the most common peak overlaps is associated with high lead concentrations, which interferes with detection of arsenic and typically results in an elevated arsenic LOD. Measured copper concentrations decrease in the presence of iron, which tends to absorb the copper X-rays. Conversely, measured chromium concentrations are enhanced in the presence of iron (EPA Method 6200 – Attachment 1). While these chemical matrix effects are generally addressed mathematically through the XRF instrument internal software, they are not eliminated. The Innov-X Handheld XRF is equipped with the internal software necessary for these mathematical corrections (i.e., measurement values generated by the FPXRF have already been corrected to account for errors resulting from chemical matrix effects; see Attachment 2).

Moisture content can affect the accuracy of both XRF measurements and laboratory analytical results. XRF measurements are more sensitive to moisture content than standard laboratory methods because XRF measures the bulk concentration of a sample's surface cross-section. When moisture content in a sample is less than approximately 15 percent, the magnitude of potential error is negligible. However, when moisture content is 15-20 percent or greater, XRF measurements are typically biased low (i.e., the reported concentration would be approximately 70 to 80 percent of what would be measured for a dry sample.

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Given the generally low moisture content of VLT materials (i.e, 4 to 7 percent moisture per the Draft Implementation Work Plan), no adverse impacts on FPXRF measurements of VLT materials would be anticipated except during or after precipitation events. Drying of samples prior to taking FPXRF measurements, if required, will be implemented to minimize or eliminate potential errors attributable to moisture effects. Sample drying will be performed in the field using a portable, handheld battery-powered not air blow dryer. Drying will be accomplished by evenly distributing the sample material in a disposable pie pan or similar flat-bottomed disposable container, then using the battery-powered blow drying to evaporate any soil moisture present. A disposable polyethylene hand trowel or plastic spoon will be used to turn and mix the sample material to facilitate complete drying of the VLT materials. Care will be taken to ensure that fine-grained materials from the sample are not dispersed from the flat-bottomed container. Drying will be considered complete when visual examination of the VLT material suggests the sample particles are dry and loose (i.e., no clumping of material is evident). The sample will then be transferred to a one-gallon plastic bag. Care will be taken to ensure that fine-grained materials from the sample are not dispersed from the flat-bottomed container.

VLT Sample Locations

The 24 locations proposed for field screening are shown in Figures 3 and 4. Two samples from each location will be collected from depths of 6-12 inches ('shallow' sample) and from 30-36 inches ('deep sample') below ground surface (bgs). The resulting 48 samples will be: 1) field-screened using the FPXRF; and 2) analyzed for metals by the project analytical laboratory (TestAmerica-Irvine) using the EPA methods shown in Table 2 in accordance with the *Quality Assurance Project Plan* (QAPP – Revision 5; ESI and Brown and Caldwell, 2009). The 20 sample locations at the north VLT borrow source area for covering the evaporation ponds are shown on Figure 3. As shown in Figure 4, four locations will be sampled at the south VLT borrow source area to represent the VLT materials proposed for the Sub-Area A cover. The relative number of sample locations is generally consistent with the total estimated cubic yards to be borrowed from each area. The sampling locations will be surveyed in the field using a handheld Trimble XT global positioning system (GPS) unit with a horizontal accuracy of +/- one meter.

Field Screening Procedures

FPXRF readings will be obtained from each sample location, as described below, using intrusive methods. Although EPA Method 6200 (Attachment 1) identifies both *in situ* and intrusive measurement methods (*in situ* mode involves direct measurement of undisturbed VLT; intrusive mode involves the collection and preparation of the VLT sample before measurement), ARC proposes to only perform intrusive measurements at each sample location to limit or eliminate potential measurement errors associated with the physical matrix effects described above. Samples will be collected using a hand auger to reach the shallow and deep sample intervals at each location. The hand auger will be decontaminated between depths at each location, and between locations, using SOPs described in the QAPP – Revision 5. Care will be taken to remove any non-VLT material from the sample.

Dry samples will be immediately transferred to the one-gallon plastic bag for the field screening process. Samples anticipated to contain moisture content values greater than the 4 to 7 percent described above will be subject to the following drying procedures: 1) remove any non-VLT materials; 2) mix and homogenize the VLT materials in the bag; 3) lay the plastic bag flat on a level non-metallic surface, and place the FPXRF probe window against the plastic bag within an area containing the homogenized material; 4) depress the trigger of the FPXRF to allow a to-be-determined minimum source count time (e.g., 120 seconds); 5) obtain measurements through the plastic bag at four locations, and average the values; and 6) record the raw measurement values and average concentration of metals on the field form (indicate non-detect values, as appropriate).

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During field screening, the field technician will visually examine the VLT to determine whether surface coatings (e.g., copper or aluminum hydroxides, 'red-dust') or other differences are present in the designated sample locations. Should visually different materials be encountered during field screening, the field technician will also collect a sample of the visually different material for characterization. Field screening of the visually different material will be performed consistent with the procedures described above for the surface and subsurface samples.

A summary of sampling activities and all FPXRF measurements will be recorded in a bound field logbook or log sheets, and entries must contain accurate and inclusive documentation of project activities. Entries will be made using permanent waterproof ink, and erasures are not permitted. Errors will be single-lined out, should not be obscured, and initialed and dated. The person making the entries will sign at the end of each day's entry, and a new page will be started for each day of sampling (QAPP – Revision 5).

VLT Sampling Procedures

Following the intrusive FPXRF measurements, the VLT samples will be transferred into laboratory-provided glass jars. The glass jars will be properly labeled using a permanent marker, with each sample label to include the following information:

- Sample identification
- Date and time of sample collection
- Sampler's initials
- Analyses requested
- Preservation method (if required)
- Project name

Chain-of-custody procedures, as specified in the QAPP-Revision 5, will be used to ensure proper handling of samples during collection and analysis and to provide sample tracking. Samples and sample documentation will be maintained in the physical possession of authorized personnel or under control in a secure area. The following information will be recorded on the chain-of-custody form:

- Sample identification number:
- Time and date of collection;
- Field sampler's name;
- Sample matrix;
- Project number;
- Any preservatives added;
- Requested analytical turn-around-time;
- Required analyses; and
- Any additional information the laboratory must know to perform the requested analysis.

The completed chain-of-custody forms will be placed in a 1-gallon water-tight zip-lock bag and, along with the samples and ice, will be sealed in the insulated container for shipment to the project laboratory (samples will be chilled to 6° F). The 48 pre-designated VLT samples, plus any samples of visually different material that may be collected, will be analyzed for the metals listed in Table 2 using EPA Methods 6010B, 6020, and 7471A (EPA, 2007).

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Quality Control Samples

In addition to the VLT samples described above, quality control (QC) samples will also be collected in accordance with provisions of the QAPP – Revision 5 to verify that sample collection, packaging and shipping are not introducing variables into the sampling chain that could provide any basis to question the validity of the analytical results or to introduce potential errors that could adversely impact accurate correlation with the FPXRF measurements. QC samples included this XRF Work Plan consist of field duplicates, equipment rinsate blanks, and matrix spike/matrix spike duplicates (MS/MSDs). All duplicate and blank samples will be labeled in the same manner as regular samples, with no indication that they are QC samples and will be submitted to the laboratory for analyses of metals using the same EPA methods as the related primary samples.

Field Duplicates

Field duplicates, to be collected a frequency of 1 per every 5 investigation samples (20 percent), will be used to check for sampling and analytical errors and reproducibility. Duplicate VLT samples will be collected by gathering twice the sample volume in a 1-gallon plastic zip-lock bag, removing any large or non-representative debris from the bag, blending the entire volume to homogenize the VLT material, and splitting the blended material into separate zip-lock bags for the primary and the duplicate samples. Splitting of the sample will be accomplished by alternately spooning portions of the blended sample into the primary and duplicate zip-lock bags. FPXRF measurements will then be taken and recorded on field forms for both samples in accordance with the procedure described above. Once the FPXRF measurements are completed, the samples will be transferred into the laboratory-provided glass jars and appropriately labeled for shipment to the project laboratory, as described above.

Equipment Rinsate Blanks

Analyses of equipment (e.g., plastic zip-lock bags and hand auger) rinsate blanks will be used to assess the efficiency of field equipment decontamination procedures in preventing cross-contamination between samples. Equipment rinsate blanks for the hand auger will be collected at a frequency of one per 20 samples (five percent, and at least once each day samples are collected, by pouring laboratory grade deionized water over the reusable sampling equipment and collecting in a clean container. One rinsate blank for each lot of plastic zip-lock bags will be submitted for analysis.

MS/MSD Samples

MS/MSD samples are investigative samples to which known amounts of analytes are added in the lab before analysis. The recoveries for spiked compounds can be used to assess how accurate the analytical method is for the site-specific sample matrix. One MS/MSD sample should be analyzed for every 20 samples (5 percent) submitted to the lab.

Quality Control Measures

The following QC measures will be implemented in accordance with Section 9.0 (Quality Control) of EPA Method 6200 (Attachment 1) to support the comparison between field screening and laboratory data:

Energy Calibration Checks

Energy calibration check samples consist of a pure element (e.g., iron, manganese, lead or copper) analyzed by the FPXRF instrument to determine whether the FPXRF is operating within resolution and stability tolerances established by the manufacturer. This check assesses whether the characteristic X-ray lines are shifting, which would indicate drift in the detector. The check also serves as a gain check in the event that ambient temperatures are fluctuating significantly more than 10 to 20° F.

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Energy calibration check will be performed in accordance with the manufacturer's recommendations. At a minimum, an energy calibration check will be performed at the beginning of each working day, after the batteries have been changed or the instrument shut off, and at the end of each field shift. The FPXRF instrument manual specifies the channel or kiloelectron volt level at which the pure element peak should appear and the expected intensity of the peak. If the energy calibration check does not meet the manufacturer's criteria, the peak obtained from the pure element spectrum will be optimized and realigned to the manufacturer's specifications using their software (Attachment 1).

Instrument Blank Samples

An instrument blank sample consists of a quartz block, silicon dioxide or a polytetrafluoroethylene block, which is used to verify that no contamination exists in the spectrometer or on the FPXRF probe window. An instrument blank sample will be analyzed at the beginning of each field shift, once after every 20th measurement during the shift, and at the end of the shift. In addition, an instrument blank sample will be analyzed whenever contamination is suspected by the field technician operating the FPXRF. If the detected concentrations exceed the established LODs, the probe window and the blank sample should be checked for contamination. If contamination is identified, the probe window should be cleaned in accordance with the manufacturer's recommendations. Lastly, once no contamination is observed, then the instrument will be zeroed according to the manufacturer's instructions (Attachment 1).

Calibration Verification Checks

Calibration verification check samples, consisting of a standard reference material (SRM) containing certified amounts of metals in VLT, will be used to check the accuracy of the instrument and assess the stability and consistency of the analysis of the target metals. Accuracy is a measure of the instrument's ability to measure the "true" concentration of an element in a sample. The check sample will be tested at the beginning and the end of each day and after every 20th measurement. The percent difference (%D) between the true value and the measured value should be less than 20 percent. If the %D is outside the acceptable range, the instrument will be recalibrated, and the batch of samples analyzed before the failed calibration verification check will be reanalyzed (Attachment 1).

Precision Measurements

Instrument precision refers to an instrument's ability to produce the same result for a number of measurements of the same sample. The precision of the FPXRF measurements will be monitored by performing several analyses of samples that contain concentrations of target metals. The first and the last sample collected each day will be subjected to precision measurements by conducting 10 replicate measurements of the sample. The precision will be assessed by calculating a relative standard deviation (RSD) of the replicate measurements for each analyte. Per EPA Method 6200 (Attachment 1), the RSD can not be greater than 20 percent for individual metals with the exception of chromium (the RSD for chromium should not exceed 30 percent). If RSD values above these thresholds for metals are observed during field screening, this variation will be noted in the field log, but no corrective actions will be taken (such RSD deviations will be subsequently evaluated during the correlation of FPXRF and laboratory analytical results following completion of the field activities described in this XRF Work Plan.

Data Evaluation and Field Characterization Approach

Laboratory analytical results obtained from the VLT samples will be compared against the FPXRF test results using a 'least squares' linear regression analysis to determine correlation coefficients for metals with appropriate LODs. Based on the correlation coefficients obtained (or not) for each of the metals listed in Table 2, ARC plans to discuss with EPA the field characterization approach and FPXRF screening method (i.e., intrusive screening) to be implemented during the removal action. The recommendations for field screening will be incorporated in the final *Removal Action Implementation Work Plan for the Evaporation Ponds*.

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References

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If you have any questions or comments, please feel free to contact me at 714-228-6774 or via e-mail at Jack.Oman@bp.com.

Sincerely,

Batchelle for

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